CENTRAL INTELLIGENCE AGENCY

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COUNTRY USSR (Kalinin Oblast)

SUBJECT Experiments on Stabilization of C-Stoff at Zavod No. 1, Podberezye

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1. C-Stoff consists of a mixture of 50 per cent by volume hydrazine hydrate with 40 to 45 per cent methanol and 5 to 10 per cent water, plus 0.3 per cent by weight of K_2CuCN_4. The German specialists at Zavod No. 1 found that it had one peculiarity, however. When exposed to the air for an indefinite period of time and without special precautionary measures, it would occasionally develop a reddish brown precipitate. This phenomenon did not occur at all times, and since there was no rocket fuel specialist among the group at Zavod No. 1 to furnish a logical explanation, it appeared that the occurrence should be investigated.

2. The study of this phenomenon began in May 1947 and lasted for approximately one year. This entire period was not devoted exclusively to work on C-Stoff, since routine chemical analyses and work on other projects had to be accomplished also. The first experiments pointed to the absorption of atmospheric oxygen as the phenomenon which was responsible for the precipitation. Samples which were sealed in test tubes by paraffined cork stoppers or rubber stoppers remained unchanged. Stratification with mineral oil or kerosene was also capable of keeping atmospheric oxygen out of contact with the C-Stoff. In all cases, the C-Stoff remained unchanged, i.e., the concentration of hydrazine hydrate did not vary. Thus, it was determined that atmospheric oxygen, aided by the presence of copper and its catalytic action, caused the oxidation of the hydrazine hydrate. It was also determined that the rapidity of the process depended upon the ratio of volumetric mass to the total area of the mass available for absorption.

For example, if test tubes containing 1, 2, 5, 10, and 20 cc. of C-Stoff were exposed to the air, the 1 and 2 cc. test tubes showed a precipitate after only two or three hours of standing, whereas the test tube containing 10 and 20 cc. C-Stoff required from three days to one week for the same process.

3. The appearance of the reddish brown precipitate (possibly pure copper or CuH_2) occurred after the concentration of the hydrazine hydrate had fallen to about 25 to 30 per cent by weight. Two means appeared capable of preventing occurrence:

   a. Addition of KCN to control the concentration of free-copper ions through a more accurate creation of the complex salt. Since the usual commercial grade of potassium cuprous cyanide used had a lower content of CN than was theoretically required by its molecular formula, it had to be assumed that free-copper ions existed in the C-Stoff solution.

      The analytical observation of C-Stoff oxidation proved that parallel to the oxidation of the hydrazine hydrate, oxidation of the cyanide salt also took place. It was noted that a continuous decrease of the CN content occurred, and apparently in concurrent reaction free copper ions appeared in the solution. Simultaneously the oxidation progressed at a greatly accelerated rate. The addition of KCN, although decreasing the speed of oxidation, led to a simultaneous decrease of combustion properties, so that this addition appeared unsuitable for power plant use. It should be noted that the addition of KCN considerably reduced the precipitation.

   b. As a second means of preventing this precipitation, a number of other substances were tested. Among these were carbon disulphide, aniline and dimethylaniline. Hydroquinone had the most effective influence, however. A certain amount of success was also achieved with KCNO and pyrocatechin. It appeared that hyroquinone was effective in even the smallest amounts (concentrations down to 0.005 per cent). An addition of 0.05 per cent was finally accepted as being most adequate. Confirmation experiments were conducted upon one liter samples exposed to atmospheric oxygen under similar conditions, that is, equal ratio of volume to surface area. At intervals of 24 hours, a small amount of C-Stoff was taken from these test specimens and analyzed. It was shown that upon addition of hydroquinone, C-Stoff remained unchanged for all practical purposes. The final C-Stoff composition, therefore, was 50 per cent to 55 per cent hydrazine hydrate, 40 per cent to 47 per cent methanol, and the remainder water with 0.3 per cent K_2CuCN_4 with 0.05 per cent hydroquinone. The actual concentration of the hydrazine hydrate and methanol was calculated each time prior to firing and depended upon the temperature which was to exist in the chamber.

4. The method used for determining the concentration of the hydrazine hydrate was by titration with iodine using a bicarbonate and starchy solution as indicator. The hydrazine hydrate used, calculated on hydrazine hydrate, gave an analytical total of 101 per cent. This high figure was caused by the presence of free hydrazine. In the initial phases of the test, hydrazine hydrate of German origin was used, but later, material of Soviet manufacture was utilized.

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this material was produced in the vicinity of Moscow. the unique manner in which the material was usually received. Orders were placed well in advance of anticipated requirements. When the material failed to arrive on the scheduled date, it was immediately reordered. When the material still failed to arrive, the German director went to the Soviet chief of the organization and informed him of the need. The Soviet chief thereafter drove somewhere, in his automobile and 50X1-HUM within a period of a few hours, trucks, bearing 200 liter aluminum drums of hydrazine hydrate, began arriving at the plant. The supply 50X1-HUM of hydrazine hydrate appeared to be plentiful, but the system of supply was based solely on Soviet whims. approximately 10 tons of the material were consumed in testing experiments.

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5. The determination of the cyanide content of C-Stoff was as follows: The fuel was diluted 1:3 of its original strength with water and added drop-wise to dilute sulphuric acid. The acid solution was placed in a retort, the opening of which was immersed in a silver nitrate solution. Heating of the acid evolved hydrogen cyanide into the $AgNO_3$. Thereafter, the silver nitrate was titrated using $Na_2Cr_2O_7$ as an indicator.

6. Determination of the copper content was as follows: C-Stoff was evaporated, the residue redissolved by nitric acid and the copper precipitated, then electrolytically or titrometrically determined.